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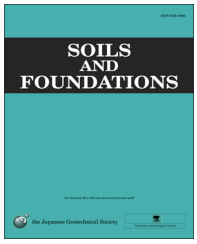

Distribution of grout material within 1-m sand column in insitu calcite precipitation technique

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Abstract

This study evaluates the potential of improving an insitu calcite grouting technique. The grout is composed of an equimolar solution of urea–$CaCl_2$ and an enzyme named urease. We examine the distribution of the grout materials and precipitated calcite within sand columns with a diameter of 5 cm and a height of 100 cm. In the first series of experiments, the concentration distributions of the individual grout materials (i.e., urea, $CaCl_2$ and urease) within the sand specimen are evaluated. In the second series of experiments, an enzyme-reagent mixed solution (i.e., grout) is injected into the sand columns to evaluate the distribution of calcite. Sand samples are collected from various vertical locations within the treated columns and the amount of precipitated calcite is evaluated. Furthermore, attempts are made to achieve the uniform distribution of the injected grout and hence uniform calcite distribution throughout the specimen. The results show that a uniform distribution of the grout materials up to a distance of 1 m from the inlet is achievable. It is also observed that a relatively uniform distribution of calcite is achievable as long as the rate of calcite precipitation is well controlled.

Keywords: Soil improvement; Grout; Precipitation; $CaCO_3$; Reaction rate

1. Introduction

A given soil may not meet the engineering requirements. The physical and mechanical properties of weak soil must be modified in order to prepare the soil for engineering purposes. Nowadays, various soil improvement techniques are being practiced.

This paper discusses an insitu mineralized calcite grouting technique. This technique employs the enzyme of urease to catalyse the hydrolysis of urea, producing ammonium and carbonate ions (Eq. (1)). The obtained carbonate ions precipitate in the presence of calcium ions (Eq. (2)). The obtained calcium carbonate is in the form of crystals of calcite (Neupane et al., 2013). The precipitated calcite forms bridges between the soil grains, binding them and restricting their movement (Harkes et al., 2010). Once deposited, the precipitated calcite is reported to be durable and to dissolve slowly on a geological time scale (van Paassen et al., 2010b).

$$CO(NH_2)_2 + 2H_2O \rightarrow 2NH_4^+ + CO_3^{2-} \quad (1)$$

$$CO_3^{2-} + Ca^{2+} \rightarrow CaCO_3 \quad (2)$$

Various works are being carried out in the field to develop insitu calcite precipitation techniques. Most of the works utilize urease producing bacteria (i.e., Sporosarcina pasteurii) to catalyse the hydrolysis of urea. The physical and mechanical properties were modified remarkably in one of the recent

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experiments, (i.e., compressive strength of up to 12 MPa) (van Paassen et al., 2010a). However, uniform improvements of the mechanical properties have yet to be achieved. The homogenous distribution of the grout material is essential for the uniform improvement of the mechanical properties. Various researchers have focussed on obtaining a homogenous distribution of the grout material and in turn, precipitated calcite. To distribute and fix bacteria homogenously in sand, Harkes et al. (2010) has suggested a two-phase injection method. In their method, a bacterial suspension is injected into the sand body immediately followed by a fixation fluid (i.e., a solution with high salt content). Al Qabany et al. (2012) studied the effect of three different concentrations (i.e., 0.25, 0.5 and 1.0 mol/L) of urea–$CaCl_2$ solution on calcite distribution over the surface of the sand grains. They observed that the calcite crystals obtained with 0.25 mol/L urea–$CaCl_2$ solution were well distributed spatially, had uniform size, and covered the contact area uniformly.

Reasons for the non-uniform distribution of the grout and hence of the precipitated calcite may vary. It may be that the control and predictability of the in-situ distribution of bacterial activities was not achieved, the reagents were unsuitable or that the resulting mechanical properties due to precipitated calcite were not sufficient (van Paassen et al., 2010b). It has been suggested that the rate of urea hydrolysis may provide a guide to understanding the chemical and biological processes, but it does not necessarily determine the rate of precipitation in the field, because several other factors are involved in the reaction in porous media and could influence the process (Al Qabany et al., 2012).

Since the uncertainties related to the bacteria do not need to be considered in the enzymatic method, it may be a relatively simple carbonate precipitation technique in comparison with the bacterial methods, as a way of achieving uniform soil improvement, (Yasuhara et al., 2012).

Upscaling the bio-mediated ground modification process from the laboratory to the field is one of the main challenges facing geotechnical engineers (Dejong et al., 2014). In this study, the insitu calcite precipitation technique for improving the mechanical properties of the soil has been evaluated with regard to the uniform distribution of precipitated calcite over distance. In our earlier small scale experiments designed to evaluate the ability of grout to improve the mechanical properties of the soil, a compressive strength of up to 1.6 MPa has obtained (Yasuhara et al., 2012). In another recent experiment carried out by our group (Neupane et al., 2015), grout was injected together with a green dye in the middle of a sand specimen (of size $\phi = 57$ cm and $H = 60$ cm) packed uniformly in drum cans. Almost uniform distribution of the injected solution was observed with a noticeable improvement in the mechanical properties. The relation between the amount of the calcite precipitated and the mechanical properties obtained in our previous experiment is shown in Fig. 1. Although the uniform distribution of the grout solution was observed, the distribution of calcite was higher near the inlet and lower away from the inlet. In the present study, the aim is to identify the cause of the non-uniform distribution of precipitated calcite.

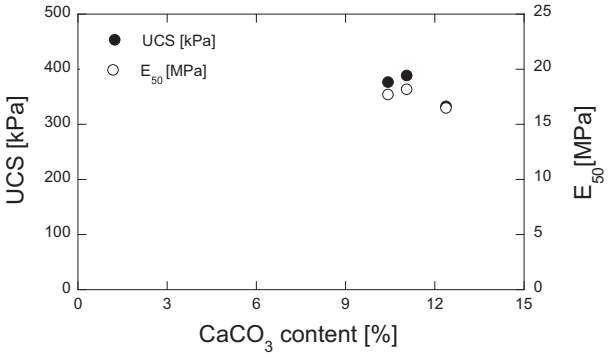

Fig. 1. Relation between calcite content and mechanical properties.

A large treatment area must be obtained to simulate the real field application of this technique. The main objective of this study is to obtain a uniform distribution of the precipitated calcite over a larger treatment area. The rate of calcite precipitation was regarded as an important factor in the controlling of the distribution of calcite. Harkes et al. (2010) concluded that a slow reaction rate is required so that all the reagents are converted. After slowing the reaction rate, calcite precipitation takes a long time and the solution can travel a longer distance before precipitation occurs. Temperature is likely to affect the rate of calcite precipitation. The rate of urea hydrolysis decreases with decreasing temperature (Cartes et al., 2009). The reaction rate decreases by factors of 2–3 for every 10 °C fall in temperature (Connors, 1990). In this study, we have made an attempt to reduce the rate of calcite precipitation by lowering the temperature of the grout materials.

2. Materials and quantification methods

The grout used in this study is a combination of reagents, i.e., urea and $CaCl_2$, and an enzyme called urease. Urea and $CaCl_2$, with claimed purity levels greater than 99%, were obtained from Kanto Chemicals Co. Inc., Tokyo, Japan and urease, with a urease activity of 2970 Units/g, was obtained from the Kishida Chemical Co. Ltd., Osaka, Japan.

Samples were prepared using two different types of silica sand, keisa 6 and keisa 8, to examine the distribution of the injected grout materials in sand with different grain sizes and different hydraulic conductivities. The physical properties of keisa 6 sand and keisa 8 sand, as obtained by Yasuhara et al. (2014) and Okamura and Inoue (2012), respectively, are shown in Table 1.

The concentration of calcium ions present in the solution was evaluated using Inductively Coupled Plasma Atomic Emission Spectrograph, ICP-AES. An ammonium ion electrode, obtained from the Toko Chemical Laboratory Co., Ltd., was used to evaluate the concentration of ammonium ions. The amount of calcite precipitated within the sand was evaluated by the CO_2 Volume Evaluation (CVE) Method (Neupane et al., 2015).

Table 1
Properties of sands.

Properties	Keisa 6	Keisa 8
D_{10} [mm]	0.200	0.040
D_{50} [mm]	0.340	0.120
C_u	1.55	2.93
G_s	2.653	2.650
$\rho_{d_{max}}$	1.713	1.554
$\rho_{d_{min}}$	1.397	1.103
k [cm/s]	0.044	0.002

Table 2
Effect of temperature on the dissociation of urea.

Exp. name	Urea [mol/L]	Urease [g/L]	Mixing temperature [°C]
TT-R	1	15	23.5
TT-L	1	15	5.0

3. Experiments

3.1. Temperature effect on rate of urea hydrolysis

Prior to the injection of the grout materials into the sand specimens, the rates of urea hydrolysis with two different mixing temperatures (i.e., 5 °C and 23.5 °C) were evaluated in PP(polypropylene) tubes. The experimental conditions are shown in Table 2. TT-R represents the mixing solution was at room temperature (i.e., 23.5 °C) and TT-L represents that at lower temperature (i.e., 5.0 °C). It should be noted that the mixing temperature indicates the temperature of the urea solution at the time of mixing. After mixing, the solution is allowed to hydrolyse at a controlled room temperature of 25 ± 1.5 °C. A solution (30 m) with final concentration as shown in Table 2 was prepared in PP tubes and allowed to react. The concentrations of ammonium ion were measured at various time intervals, i.e., 5 min, 10 min, 30 min, 1 h, 2 h, 5 h and 25 h after mixing.

The evolutions of the ammonium ion concentration obtained from the PP tube scale experiments are shown in Fig. 2. As is apparent, the evolution of the ammonium ion concentration in case TT-L (i.e., mixed at 5 °C), was lower than that for TT-R (i.e., mixed at room temperature) up to a time interval of 30 min. After 1 h, the concentrations of ammonium ions measured in both samples was almost equivalent, and the ammonium ion concentration ranged between 85% and 90% of the theoretical maximum value.

3.2. 1-m sand column experiments

Experiments were carried out to determine whether the uniform distribution of the precipitated calcite over a distance of about one meter from the injection is possible. An acrylic pipe, with an inner diameter of 5 cm and a length of 100 cm, was utilized. The distribution of the grout materials and the precipitated calcite throughout the sand specimens packed inside the acrylic pipe were evaluated.

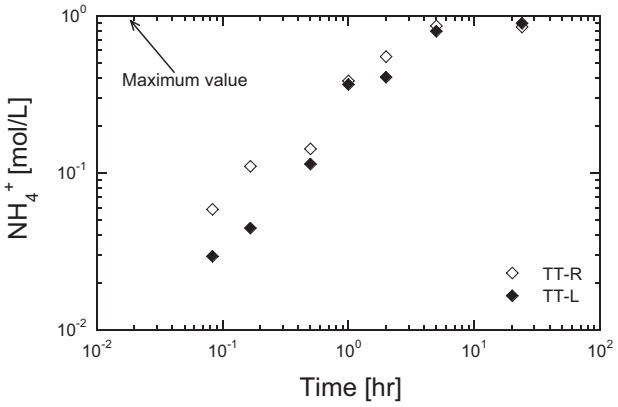

Fig. 2. Effect of mixing temperature on the evolution of ammonium ion.

Two sets of experiments were conducted. The first set of experiments was performed to examine whether the distribution of individual grout materials was uniform throughout the sand specimens or not. The second set of experiments was conducted to examine the distribution of the precipitated calcite within the sand specimens. Individual grout materials were injected in the first set of experiments, whereas mixed grout was injected in the second set of experiments.

3.3. Experimental setup and procedure

A schematic diagram of the 1-m sand column experiments is shown in Fig. 3. The same acrylic pipe was used for all experiments. The acrylic pipe contains removable caps with porous stones at both ends. The porous stones prevent the direct contact of the water/solution flow to the sand which in turn helps to prevent the change in relative density. It also helps to distribute the solution uniformly throughout the specimen. Both of these caps are connected to separate tubes, with a diameter of 8 mm, to inject and to drain the excess grout. The acrylic pipe contains 11 holes with a diameter of 2 mm at equal intervals of about 10 cm centre-to-centre. These holes are kept covered with silicone seals, and are utilized to pump the solution sample at a later stage.

The experimental procedure described in more detail in Neupane et al. (2014). At first, the bottom cap of the acrylic pipe was kept closed. All the holes were covered with silicone seals. Sand was poured into the pipe in 5 layers, maintaining a relative density of about 50% in each layer. Two pore volumes (PV) of water were injected from the bottom to the top to remove air from the specimen to prevent the formation of air pockets during solution injection. However, the degree of saturation was not measured in this work. Afterwards, one pore volume (i.e., 800 mL and 1200 mL in case of coarser and finer soil respectively) of freshly prepared injecting solution was injected.

The properties of the injected solutions used in the first set of experiments are shown in Table 3. The temperature of all solutions and sand was 25 ± 1.5 °C. $CaCl_2$ was injected in coarser and finer soil as represented by CaC and CaF,

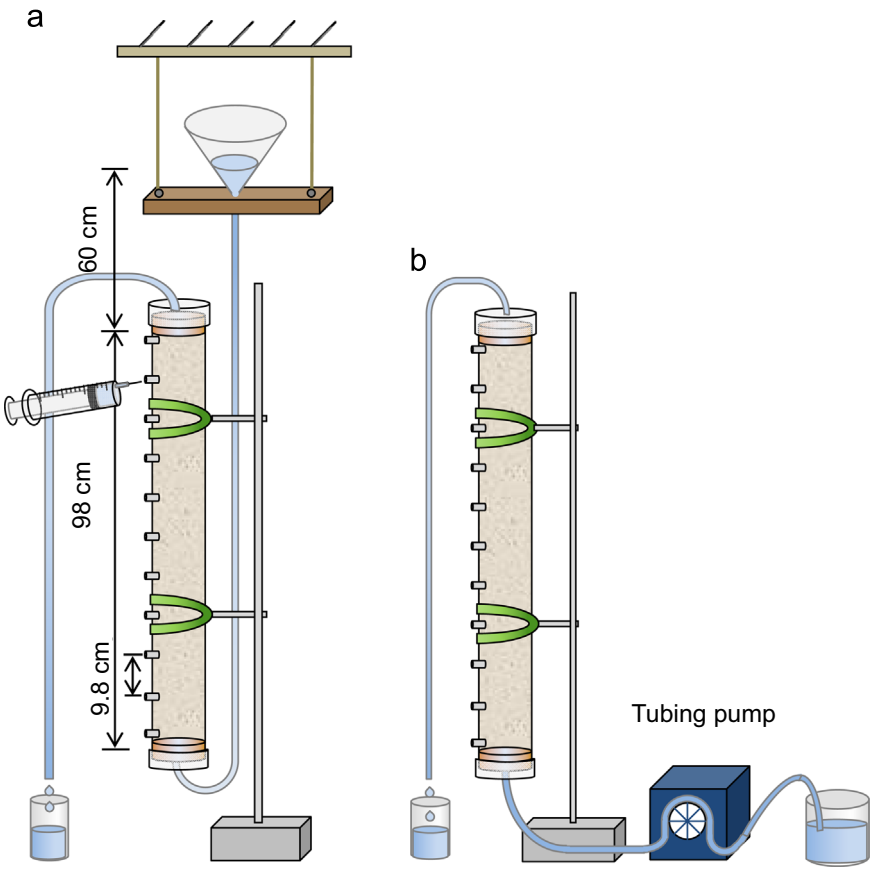

Fig. 3. Schematic of the 1-D flow experiment (a) keisa 6, (b) keisa 8.

Table 3
Conditions for 1-m sand column experiments for the evaluation of distribution of individual grout material.

Exp. name	Injecting solution	Injection volume [PV]	Head [cm]	Sand type	Dilution	Mixing solution	Evaluation method
CaC	1 mol/L $CaCl_2$	1.0	60	Keisa 6	NA	NA	ICPAES
CaF	1 mol/L $CaCl_2$	1.0	Pu	Keisa 8	NA	NA	ICPAES
UaC	1 mol/L Urea	1.0	60	Keisa 6	10 Times	30 g/L Urease	Ion meter
UaF	1 mol/L Urea	1.0	Pu	Keisa 8	10 Times	30 g/L Urease	Ion meter
EL1	15 g/L Urease	1.0	60	Keisa 6	10 Times	1 mol/L Urea	Ion meter
EH1	15 g/L Urease	1.0	80	Keisa 6	10 Times	1 mol/L Urea	Ion meter
EH1.5	15 g/L Urease	1.5	80	Keisa 6	10 Times	1 mol/L Urea	Ion meter
EP1.5	15 g/L Urease	1.5	Pu	Keisa 8	10 Times	1 mol/L Urea	Ion meter

*NA – Not applicable ** Pu – solution is injected using a pump.

respectively, to evaluate the distribution of the calcium ion concentration. Similarly, UaC and UaF represent the injection of urea to evaluate its distribution in coarser and finer soil, respectively. In the case of CaC, CaF, UaC and UaF, a constant head of 60 cm was maintained to adjust the flow rate to about 60 mL/s. The flow rate was kept constant throughout these experiments because precipitation reaction does not take place in the case of individual solution injection. EL1 and EH1 represent the injection of 1.0 PV of enzyme solution at a lower head (i.e., 60 cm), and a higher head (i.e., 80 cm), respectively, in coarser sand. EH1.5 represents 1.5 PV of the enzyme solution at the higher head (i.e., 80 cm) in coarser sand. EP1.5 represents the injection of 1.5 PV of enzyme solution in finer sand. As the permeability of finer soil is very low, peristaltic pump (Fig. 3) was used to maintain a similar flow rate.

The properties of the injected solution for the second set of experiments are shown in Table 4. The experimental conditions of EH1.5 were replicated in these set of experiments, including the concentration of urea and the use of an injection head. Two experiments, Cal-L and Cal-R, were conducted. The concentration and the volume of the grout were kept equal (i.e., 800 mL) in both cases. The solution was prepared in water at a room temperature in case of Cal-R, whereas, in Cal-L, the temperature of the prepared solutions was lowered to 5 °C separately before mixing. The sand specimen preparation

Table 4
Conditions for 1-m sand column experiments for the evaluation of the distribution of calcite.

Exp. name	Urea–$CaCl_2$ [mol/L]	Urease [g/L]	Head [cm]	Inj. vol. [PV]	Mixing temperature [°C]	Sand
Cal-R	1.0	15	80	1.5	23.5	Keisa 6
Cal-L	1.0	15	80	1.5	5.0	Keisa 6

and injection were conducted in a temperature controlled room (25 ± 1.5 °C) for both experiments. The 80 cm head applied in this set of experiments was found to maintain a constant flow rate of about 80 mL/min throughout the experiment. This is because the precipitation starts to appear about 30 min after mixing the grout solutions, whereas the injection of 800 mL solution takes only about 10 min.

3.4. Sample collection

Two types of samples were collected. To evaluate the distribution of the injected grout materials (i.e., cases CaC, CaF, UaC, UaF, EL1, EH1, EL1.5 and EP1.5), liquid samples were collected just after completing the solution injection. A total of 5-mL of solutions were pumped out of every hole (i.e. at 10 cm centre-to-centre) with the help of syringes fitted with a needle. The sample solutions for cases CaC and CaF were subjected to ICP-AES to evaluate the concentrations of calcium ion present in the solution. The samples collected from the other cases (UaC, UaF, EL1, EH1, EL1.5 and EP1.5) were mixed with an equal volume of the mixing solution, as shown in Table 3. The mixed samples were allowed to react in closed PP tubes for 24 h. After 24 h, 1 mL of the mixed sample was diluted with 9 mL of deionized water, and the ammonium concentration of each of these samples was evaluated.

To evaluate the distribution of calcite throughout the specimens (i.e., cases Cal-L and Cal-R), injected grout was allowed to react inside the acrylic pipe for 24 hours. After 24 h, the column was flushed with 2 PV (i.e., 1600 mL) of deionized water. The caps were removed and about 20 g of sand samples was collected at every 10 cm. The collected samples were dried in the oven and the amount of precipitated calcite was evaluated.

4. Results and discussion

4.1. Distribution of individual grout materials

The vertical distributions of the calcium ion for cases CaC and CaF are shown in Fig. 4. As is apparent in the figure, the concentrations of calcium ion were almost uniform throughout the sand specimens. The vertical distributions of ammonium ion for cases UaC and UaF are shown in Fig. 5. As is observed in the figure, the majority of the ammonium ion concentrations in the case of UaC was nearly equal to the ammonium ion concentration of the solution before injection (i.e., concentration at 0 cm). In the case of UaF, the ammonium ion

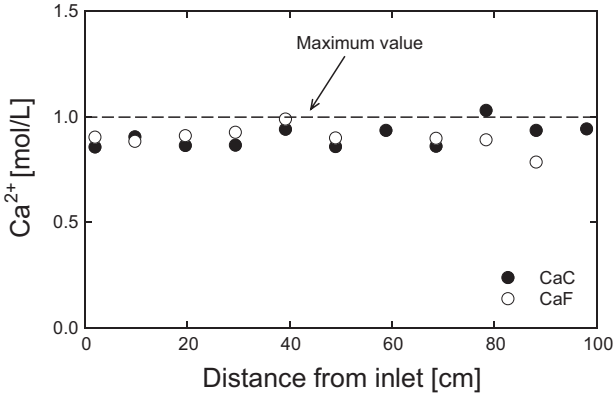

Fig. 4. Vertical distribution of the calcium ion obtained from the injection of $CaCl_2$.

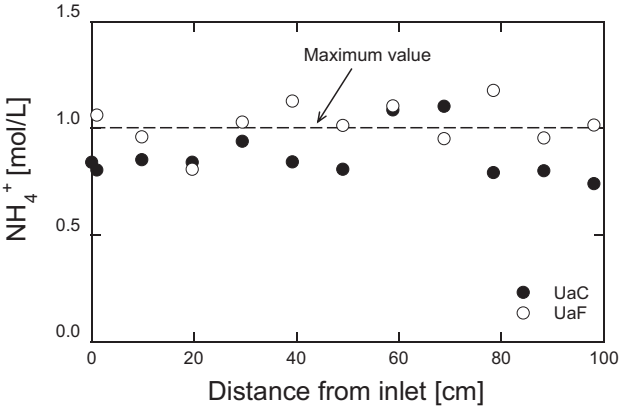

Fig. 5. Vertical distribution of the ammonium ion obtained from the injection of urea.

concentrations oscillate about a mean value of 1.0 mol/L. The distribution of ammonium ion for case UaC is more uniform than that for case UaF. The relatively higher variation in urea concentrations in case Ua2 may be due to the smaller pore size of keisa 8 sand.

The urease content was evaluated indirectly. The urease content was evaluated by measuring the ammonium ion concentration obtained from the reaction of the collected urease samples with an equal volume of concentration fixed urea solution. The vertical distribution of ammonium ion for cases EL1 and EH1 is shown in Fig. 6. As shown in the figure, the concentration of ammonium ion was uniform up to a length of 60 cm, and then began to decrease. The decrease in ammonium ion concentration in EL1 was abrupt, while that in case EH1 was relatively gradual. The distribution of ammonium ion was observed as relatively uniform in the case of EL1.5 when the injection volume was increased by 50% (i.e., 1.5 PV). Afterwards, an equal pore volume of the solution was injected into keisa 8, as is represented by case EP1.5. As shown in Fig. 6, the urease content was noticeable only up to a length of 10 cm from the inlet. The urease content suddenly drops to a very low value at a distance of 20 cm and remains low at further distances. This may be due to the smaller pore size of keisa 8 sand. The small pores may block and absorb the colloidal particles of urease at short distances.

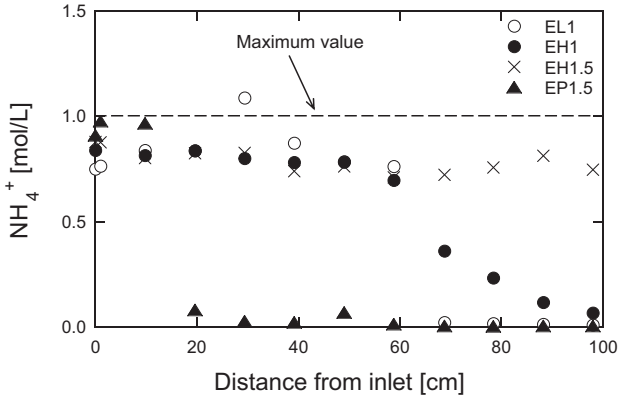

Fig. 6. Vertical distribution of the ammonium ion obtained from the injection of urease.

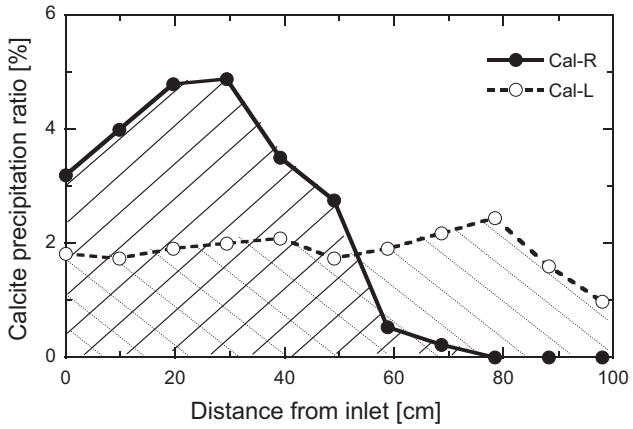

Fig. 7. Vertical distribution of the precipitated calcite obtained from the injection of grout materials.

These results show that relatively uniform distributions of urea and $CaCl_2$ over a distance of almost 1 m for both finer and coarser sands are achievable. The uniform distribution of urease is also easily achievable in coarser sand with a 50% increase in the injection volume. Since all three grout materials can be distributed uniformly throughout a distance of about 1 m from the injection, the treatment volume of 2 m in diameter may be achievable in the real field. The next step of this work is to achieve a uniform distribution of the precipitated calcite.

4.2. Distribution of calcite

The vertical distribution of the calcite within the sand column is depicted in Fig. 7. The calcite precipitation ratio is the ratio of the mass of the precipitated calcite to the mass of sand, expressed in percentage. The solid and open circles represent the calcite distributions at mixing temperatures of 23.5 °C (i.e., Cal-R) and 5.0 °C (i.e., Cal-L), respectively. As was observed in the figure, the calcite in the case of Cal-R was concentrated close to the injection. The calcite precipitation ratio reached its highest value at a distance of 30 cm from the inlet and began to decline to low values after 50 cm. On the other hand, a uniform distribution of calcite was observed over a distance of about 90 cm in the case of Cal-L. A noticeable amount of calcite was observed even at a distance of 98 cm from the inlet. A relatively non-uniform calcite distribution in the case of Cal-R may be due to the faster rate of calcite precipitation. A relatively uniform calcite distribution in the case of Cal-L, may be due to the reduced precipitation rate because of the prescribed lower temperature of the mixing solution.

As the shaded portion in Fig. 7 indicates, the total amount of precipitated calcite in the case of Cal-R is higher than that in the case Cal-L. The precipitated calcite was observed only up to a distance of 80 cm in the case of Cal-R. It can be concluded that all the attainable calcite was precipitated within that distance. Conversely, in the case of Cal-L, an appreciable amount of calcite was observed even near the outlet of the pipe. It is possible that some amount of calcite may have precipitated beyond the specimen, i.e., from the drained solution. This suggests that a treatment area of more than 2 m in diameter may be achievable during the real site application.

The method of reducing the precipitation rate by lowering the temperature was observed to help achieve the relatively uniform distribution of calcite. The higher amount of calcite close to the injection and the lower amount of calcite away from the inlet observed in our previous works was possibly due to the faster rate of calcite precipitation.

5. Conclusions

Ensuring a uniform distribution of precipitated calcite is a great challenge of insitu calcite precipitation technique. In this study, the distribution of the individual grout materials within two types of sand soil (i.e., coarser and finer) was examined. The uniform distribution of the reagents was obtained in both the finer and coarser sands evaluated in this work. However, the uniform distribution of urease enzyme was only obtained in the coarser sand. After achieving the uniform distribution of the individual materials, grout (i.e., the solution of urea–$CaCl_2$ and urease) was injected into the coarser sand. The distribution of the precipitated calcite within the specimen was examined for grouts mixed at two different temperatures. The mixing temperature was shown to have a noticeable effect on the treatment distance. Grout with a higher mixing temperature precipitated such that a higher amount of calcite was observed close to the injection point. On the other hand, grout with a lower mixing temperature contributed to the relatively uniform calcite precipitation throughout. The findings of this indicate that it may be possible to obtain a uniform calcite precipitation up to a treatment distance of 1 m from the injection as long as the rate of calcite precipitation is controlled.

Acknowledgments

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